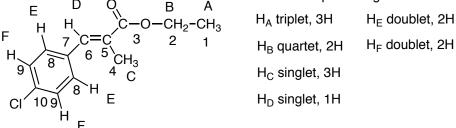
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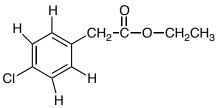
Quiz 1, 50 pts, Spring, 2012

1. For the following molecules identify (a) the number of proton signals, their splitting patterns and their relative intensities (b) the number of carbon signals. (15 pts)

There are 6 proton signals and 10 carbon signals.



2. Identify the following molecule of formula $C_{10}H_{11}ClO_2$; IR: 1735 cm⁻¹; ¹H: δ 1.2, triplet, 3H; 2.8, singlet, 2H; 3.9, quartet, 2H; 7.2, doublet, 2H; 7.4, doublet, 2H. (15 pts)

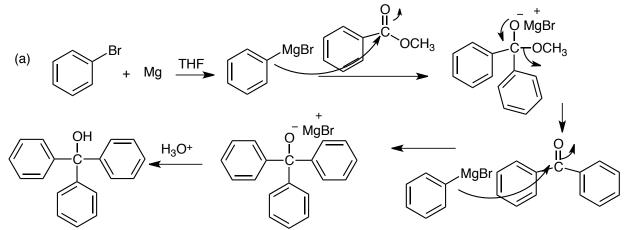


3. Answer the following questions on the preparation of acetyl salicylic acid from salicylic acid, acetic anhydride and phosphoric acid. (a) Is salicylic acid soluble in acetic anhydride? Explain briefly. (b) Why is it important to keep the spatula in the test tube at all times during recrystallization? (c) One student used a large test tube during the recrystallization instead of a small test tube and ended up adding about 20 mL of the 50:50 ethyl acetate/hexane solvent. All of her solid dissolved in the hot solution but when she cooled it down to room temperature and then in an ice bath she did not get any crystals formed. Was her experiment ruined? What could she do to recover her product? (7 pts) (a) The salicylic acid is not soluble in acetic anhydride at room temperature. The solution on initial mixing remained cloudy and only became clear on heating as the salicylic acid reacted to form the acetyl salicylic acid. (b) The spatula is for stirring the solution and more importantly it acts as a boiling chip. This is critical when doing a recrystallization using a test tube to prevent the solution from boiling over out of the test tube into the water bath. (c) Not her experiment was not ruined. She should reduce the volume by evaporative heating.

4. In the preparation of triphenyl carbinol from bromobenzene, magnesium and methyl benzoate ($C_6H_5CO_2CH_3$) (a) show the overall reaction, including all intermediates. (b) What is the purpose of the drying tube? (c) How can you tell that you have formed the organomagnesium (Grignard) reagent? (d) What is the purpose of adding the aqueous HCl at the end of the reaction? Show the reaction that occurs. (e) Do you need to have the drying tube attached to your reaction flask at this point? Explain briefly. (13 pts)

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(b) The drying tube absorbs moisture from the air but allows the pressure inside the flask to equilibrate with the outside atmosphere.

(c) You can tell when the reaction is finished when most or all of the magnesium chips have been consumed to make the Grignard reagent.

(d) The purpose of the aqueous HCl at the end of the experiment is to protonate the oxygen anion to make the alcohol.

(e) The drying tube is not needed at this point because you are adding water along with the acid.